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EXTRUSION RHEOMETRY OF FLUID MATERIALS.(U)

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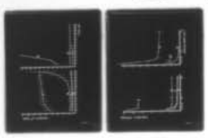

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EXTRUSION RHEOMETRY OF FLUID MATERIALS

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Short Title

Extrusion Rheometry

78 06 12 158

Synopsis

A capillary extrusion rheometer was developed and tested using Newtonian oils of known viscosity and seven endodontic sealers. The described apparatus and technique appears appropriate for comparative assessment of the rheological characteristics of a variety of fluid dental materials.

EXTRUSION RHEOMETRY OF FLUID MATERIALS

The ability of fluid-substances to conform to the shapes of substrate surfaces or to gain entrance to minute spaces is governed largely by rheological phenomena. An understanding of rheological behavior in general, and of flow properties in particular, is essential to the rational selection and clinical application of certain dental materials. Unfortunately, the expense of complex testing machines (rheometers) and the inherent inadequacies of available instrumentation of simpler design have discouraged the pursuit of research in this field.

In the present work, comparative assessment of the rheological behavior of fluid-materials was made through the use of a recently developed extrusion rheometer.

Materials and Methods

The test instrument exhibited design features that allowed (1) use of amounts of materials commensurate with those employed in clinical practice, (2) monitoring of changes in viscosity with time, (3) definition of the shear rate dependence of viscosity (4) control of temperature and (5) use of auxillary equipment common to materials' testing laboratories.

The instrument's water jacket, base and reservoir housing were machined from brass. A diagram depicting the relationship of these components to one another is shown in Figure 1.

The water jacket was 9.5 cm in height with internal diameters of 2.5 cm and 0.63 cm that accommodated the reservoir housing and capillary, respectively. Three rods (dia 1 cm) were attached to the top and bottom plates of the water jacket to provide additional support during the extrusion process. The water jacket was attached to a hollow base 7.5 cm in diameter. An opening in the base permitted observation of the extrudate during the experimental trials.

The reservoir housing was a 4-cm segment of 0.8-cm inside diameter brass tubing. One end of the housing was fitted with a lock-nut and internal "O" rings to provide a positive seal for attachment of the capillary. The loadbearing surface of the housing was radiused and matched to the top plate of the water jacket to allow self alignment of the housing during the period of load application.

The reservoir (ID 3.9 mm) and plunger (dia 4.0 mm) were obtained by modification of a conventional 0.5 cc glass hypodermic syringe.[*] A 3.2-cm segment was removed from the center portion of the syringe barrel and stabilized in the reservoir housing with an autopolymerizing resin.[+]

Blunt-end hypodermic needles[#] (ID ~0.52 mm and length 55 mm)

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* "Perfectum" Tuberculin Syringe 1/2 cc, Popper and Sons, Inc., New Hyde Park, NY 11040.

+ Kadon, L. D. Caulk Co., Milford, DE 19963.

"Perfectum" Bio Medical Needles, 21 Gauge, Popper and Sons, Inc., New Hyde Park, NY 11040.

were used as disposable capillaries. A new capillary was used for each test-run.

Test materials included three Newtonian fluids of known viscosity[§] and seven endodontic sealers.[¶]

The endodontic sealers were manipulated in accordance with their respective manufacturer's instructions to yield volumes of about one cubic centimeter. Following placement of a material into the reservoir, the plunger was depressed by application of thumb pressure until discharge of the extrudate from the distal orifice of the capillary was observed. The filled reservior and capillary were placed within the water jacket. Then the entire assembly was positioned on a constant strain-rate testing machine[**] to allow loading of the plunger in an axial direction.

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§ Viscosity Standards, 30,400 cp; 57,000 cp; and 100,000 cp Brookfield Engineering Corp., Stoughton, MA 02074.

¶ Materials A, B and C, Roth Cements, types 601, 801 and 811, respectively, Roth Drug Co., Chicago, IL 60610; material D, Diaket, Premier Dental Products Co., Norristown, PA 19401; material E, Tubli-Seal, and material F, Kerr Pulp Canal Sealer, Kerr Sybron Corp., Romulus, MI 48174; material G, Proco-Sol, Proco-Sol Chemical Co., West Conshohocken, PA 19428

** Instron Universal Testing Machine, Instron Corp., Canton, MA 02021.

The test substances were extruded through capillaries at crosshead speeds of 0.02, 0.05, 0.10, 0.20 and 0.50 inch per minute. Five trials were made with each material at each crosshead speed. The load required for extrusion was measured as a function of time. Extrusion of the endodontic sealers was begun one and one-half minutes following the end of mixing. Initial force values were recorded 30 seconds later.

Tests using fluids of known viscosity were accomplished at 25±1C, whereas those using endodontic sealers were made at 37±1C.

The load-time data were transformed into the rheological quantities of shear stress (τ) and shear rate (γ) by substitution of load values, crosshead speeds and measured dimensions of the plunger and capillary into appropriate equations for the mechanics of flow in capillaries.[3] Viscosities were calculated from a version of the Hagen-Poiseuille equation. Classification of the rheological behavior of the specimens was accomplished by application of a power law equation: $\tau\text{-}K(\gamma)^n$ where τ is the shear stress at the capillary wall, γ the shear rate, and K and n are constants. Calculated values of $n = 1$; $n<1$ or $n>1$ permitted classification of the test material as Newtonian, pseudoplastic or dilatant, respectively.

Results

The selected crosshead speeds produced shear rates ranging from 8 to 205 reciprocal seconds (sec^{-1}).

Data obtained on extrusion of materials of known viscosity

yielded values of 318±30 poise, 546±34 poise and 942±110 poise for the 304, 570 and 1,000 poise standards, respectively. Solution of the power law equation by regression analysis of plots of log shear stress versus log shear rate for the low, middle and high viscosity standards gave respective "n" values of 1.00, 0.98 and 0.93.

The effect of time on the viscosity of the endodontic sealers is illustrated in Figure 2. Six materials exhibited increased viscosity with time. One sealer, material G, demonstrated no change in viscosity over the 15-minute period of measurement. Initial viscosities at a shear rate of 21 sec^{-1} ranged from 55 poise for material B to 4,300 poise for material D.

Shear-rate dependence of the viscosity of the endodontic sealers is shown in Figure 3. Viscosity at a shear rate of 205 sec^{-1} ranged from 37 percent (material A) to 69 percent (material B and C) less than that observed at 8 sec^{-1}. Calculated "n" values ranged from 0.04 (material E) to 0.89 (material A).

Discussion

Several factors bracket the range of viscosity over which satisfactory employment of the extrusion rheometer could be anticipated. Inherent sensitivity of the load measuring device and the magnitude of the frictional force developed within the evacuated reservoir impose a minimum measurable limit. On the other hand, strength and rigidity of the test assembly as well as relaxation of the rheometer-load delivery couple delineate the maximum limit of measurable viscosity.

Observed viscosities of the three Newtonian standards exhibit variations that are compatible with measured discrepancies (0.02 mm) in the diameters of the capillaries. Additionally, it is recognized that some random errors associated with the reported data are manifestations of leakage of material past the plunger, entrapment of air bubbles in the test fluid, intermolecular friction, fluid compressibility and capillary end-effects.

The described procedure appears to be suitable for comparative assessment of the rheological properties of endodontic sealers. Derived "n" values indicate that the sealers are pseudoplastic and that their viscosity decreases with increasing rate of shear. Results obtained by the extrusion method agree with those obtained by other viscometric techniques.[4]

Conclusions

Comparative assessment of the flow properties of selected fluid materials was made by a capillary extrusion technique. The Newtonian character of oils of known viscosity and the pseudoplastic behavior of endodontic sealers was demonstrated.

Legends for Figures

Figure 1. Capillary extrusion rheometer.

Figure 2. Viscosity of endodontic sealers as a function of time at a shear rate of 21 reciprocal seconds.

Figure 3. Viscosity of endodontic sealers as a function of shear rate two minutes after the end of mixing.

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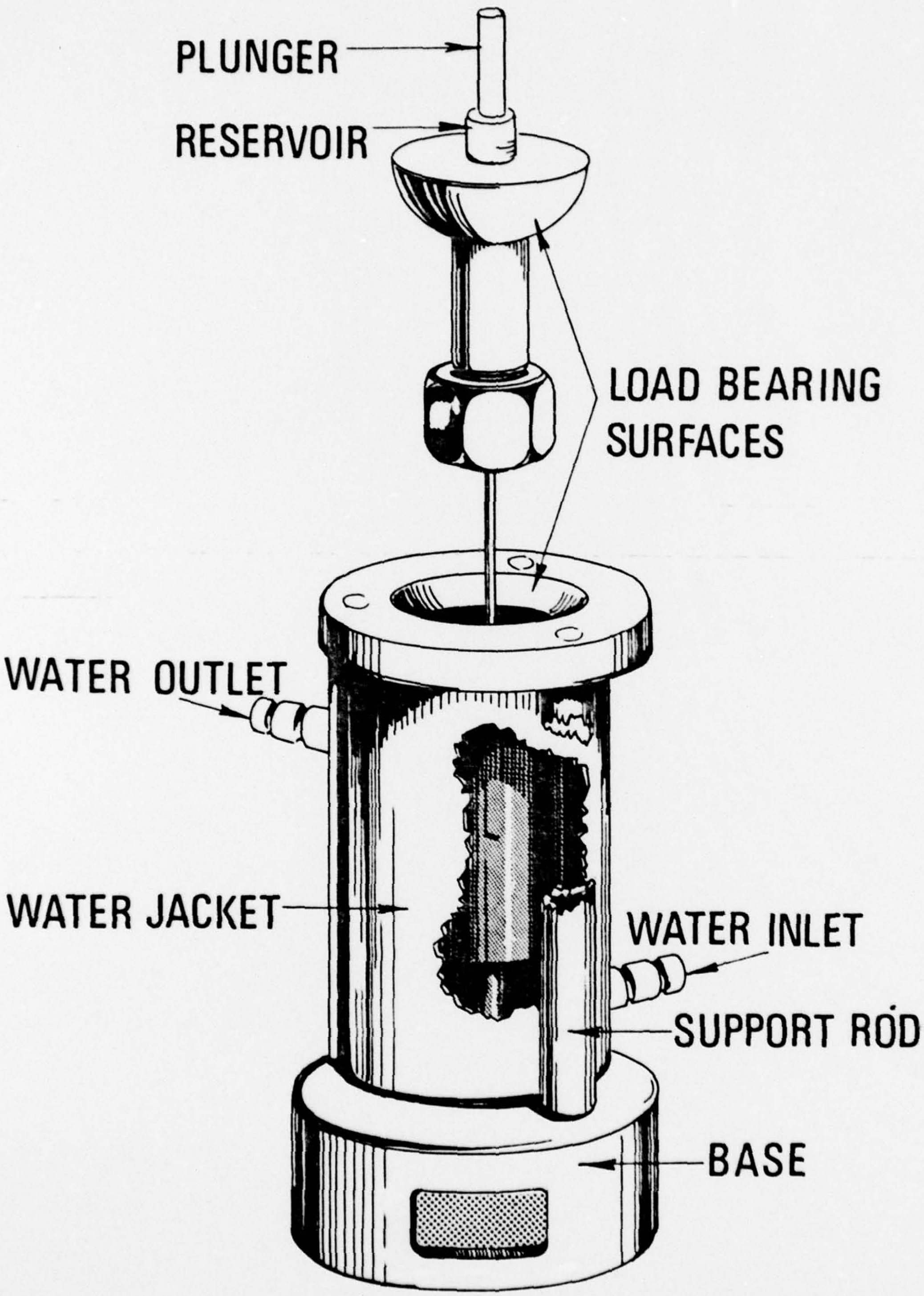
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RESERVOIR
LOAD BEARING SURFACES
WATER OUTLET
WATER JACKET
WATER INLET
SUPPORT ROD
BASE

Figure 1

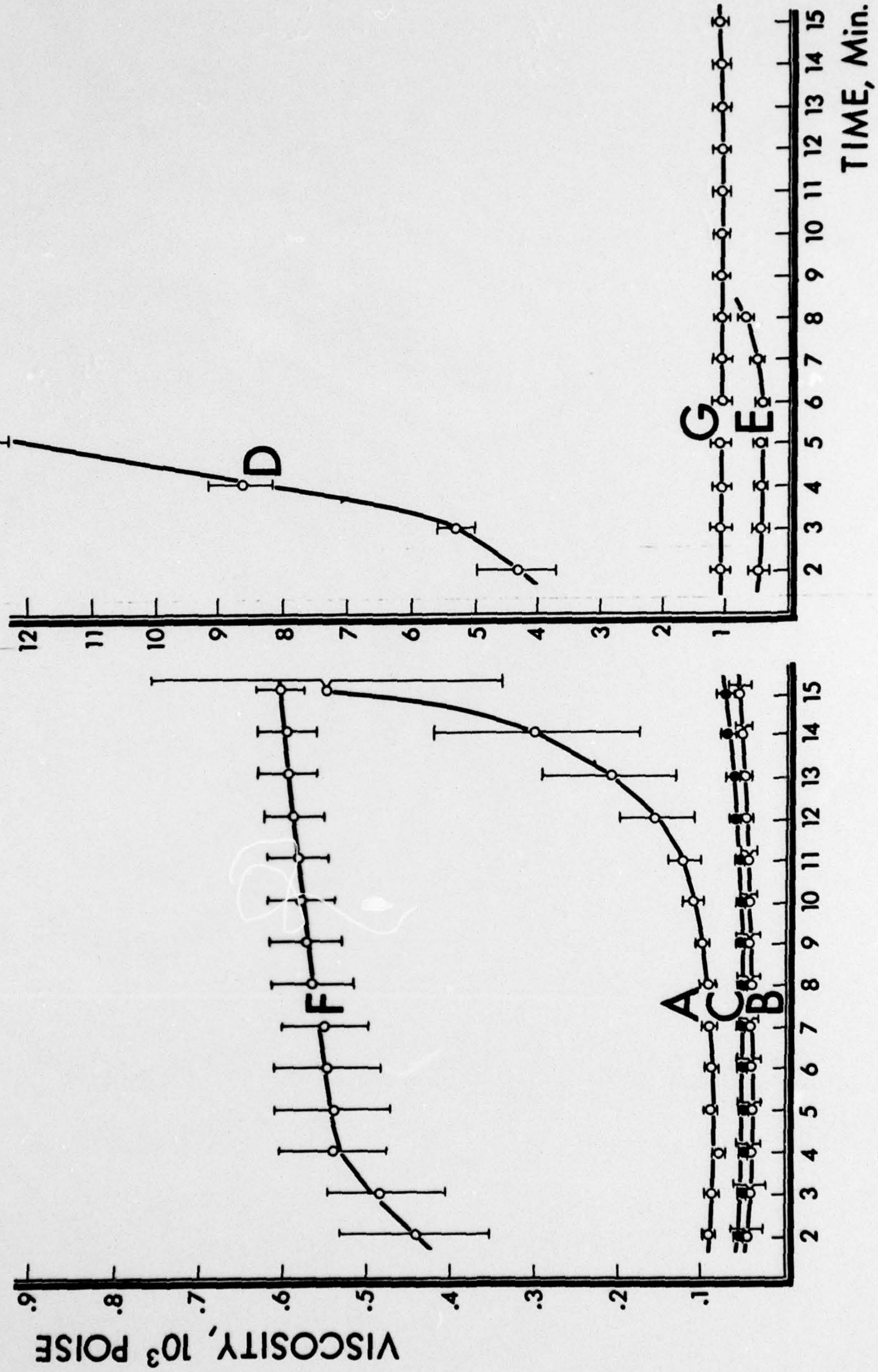
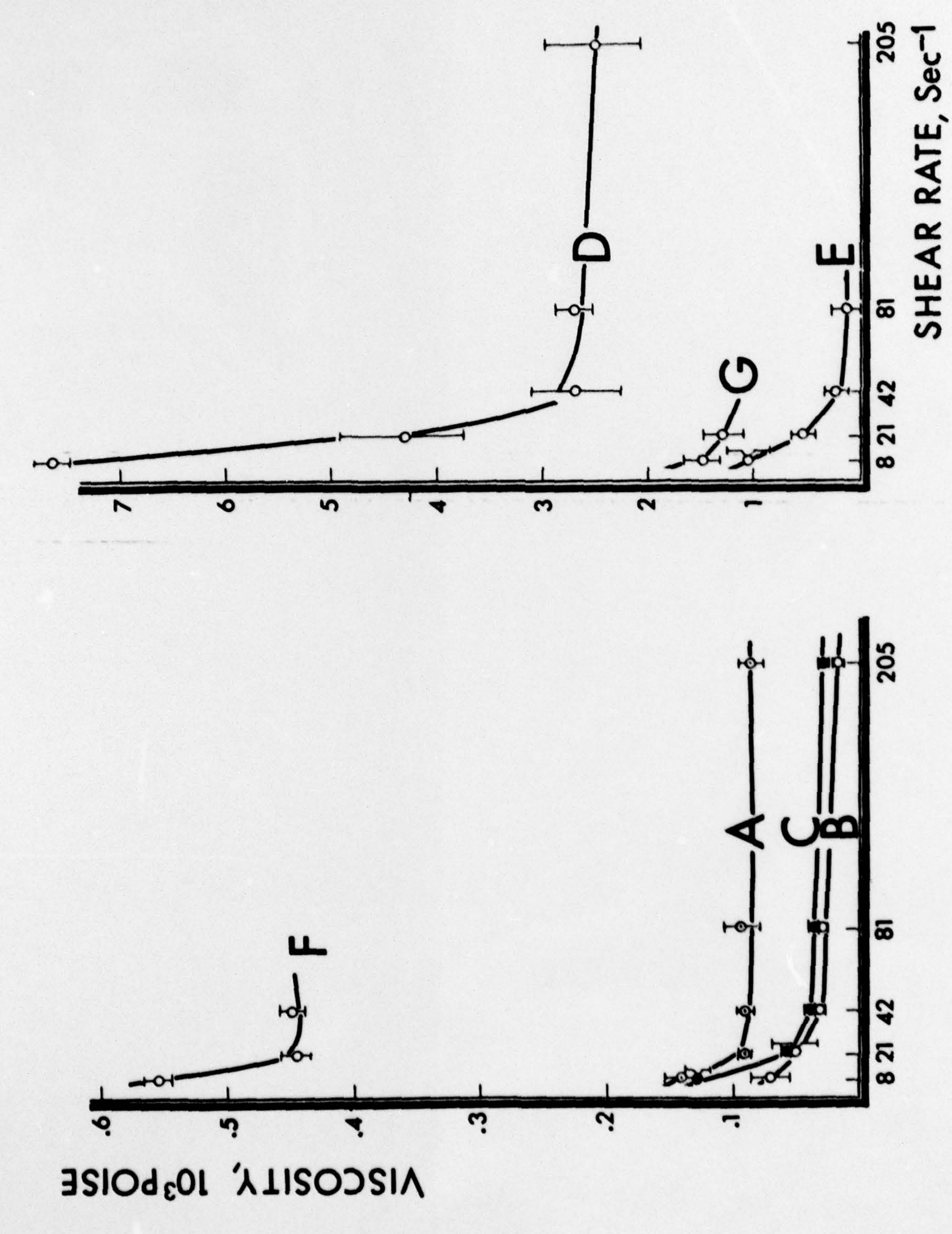


Figure 2


VISCOSITY, 10^3 POISE
SHEAR RATE, Sec^{-1}
A
B
C
D
E
F
G
8 21 42 81 205
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1 2 3 4 5 6 7

Figure 3